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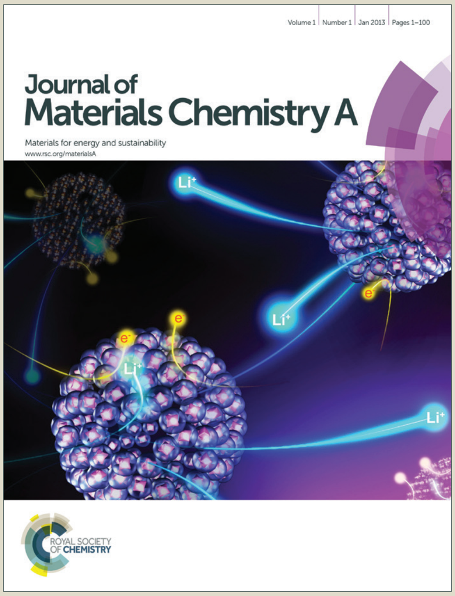

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ARTICLE

Pyrogallic acid coated polypropylene membrane as separator for lithium-ion batteries

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To solve the wetting capability issue of commercial polypropylene (PP) separators for lithium-ion batteries (LIBs), we developed a simple and new dipping surface coating method. In this method, pyrogallic acid (PA) is used as the sole coating precursor, and the PA coatings are formed spontaneously onto the PP separator surfaces. Attenuated total reflection-infrared spectra (ATR-IR) and X-ray photoelectron spectroscopy (XPS) measurements show that the PA has been successfully coated on the surface of the PP separator. The PA coatings make the PP surfaces hydrophilic, while the micro-porous structure of the separators remains intact. The improved LIBs performance including discharging specific capacity, cycling performane and rate capability are obtained by using these PA coated PP separators due to their better wetting capability, higher electrolyte uptake and ionic conductivity. XPS measurements indicate that the PA coatings show good stability and strong adhesion onto the PP separators after cycling test in LIBs. This study provides an effective and cheap way to achieve hydrophilic modification of commercial polyolefin separators for LIBs applications, and has potential application to solve other membranes with similar surface property issues.

Introduction

Lithium-ion batteries (LIBs) are expected to be used more widely for hybrid and completely electric vehicles due to their high energy density and high open-circuit voltage.[1,2] However, their performance including cycle lives, energy and power densities, and safety concerns have been not able to completely meet the requirements of efficiency storage and/or powering for hybrid or electric vehicles. In order to meet these requirements, the performance of all the battery components including electrodes (cathode and anode), electrolytes (liquid, polymer or solid state electrolytes) and separators needs to be further improved. Recently, extensive research has been denoted to electrode materials and electrolytes,[3-6] however, less attention has been paid to the improved separator technologies even though they are also critical to the battery performance. Now, commercial available polyolefin (polyethylene [PE] and polypropylene [PP]) microporous films have been widely used as the LIB separators due to their low cost, high excellent mechanical strength, good electrochemical stability, proper micro-pore structure and thermal shutdown properties.[7] However, these polyolefin separators have hydrophobic surface characters and low surface energies, which cause these separators to exhibit poor compatible with conventional liquid electrolytes such as ethylene carbonate (EC) and dimethyl carbonate (DMC) based liquid electrolytes.[7] The poor wetting capability of these polyolefin separators seriously hinders the absorption and diffusion of the liquid electrolytes within the separators, which could directly affect the cycle stability and power performance of LIBs.

To solve the poor wetting capability issue of these commercial polyolefin separators, various modification approaches have been tried to improve the hydrophilic surface of these separators. Some hydrophilic monomers such as acrylonitrile,[8] acrylic acid,[9] diethylene glycol-dimethacrylate,[9] methyl methacrylate,[10] glycidyl methacrylate[11] and 2,4,6,8-tetramethyl-2,4,6,8-tetravinylcyclote-trasiloxane,[12] have been grafted onto the separator surfaces by using plasma treatment,[8] ultraviolet irradiation,[13] gamma irradiation[14] and electron beam irradiation grafting processes.[9-12] In addition, various hydrophilic polymers such as poly(ethylene oxide),[15] poly(methyl methacrylate)[16] and polyimide[17] have been coated onto the separator surfaces with solution process. However, these methods also generate some negative effects, such as pore blocking and porosity decreasing resulting in decrease the high-power capability,[18,19] multi-step operations with high manufacturing cost, environmental problems due to the use of toxic organic solvents, and the requirements of complex or sophisticated equipment. [18,19]

Inspired by the versatile adhesion capability of polydopamine, recently Choi et al. coated polydopamine on commercial PE separators through dopamine self-polymerization in weak alkaline aqueous solution, and found that the polydopamine-coating turned PE separators more hydrophilic and improved the power performance of battery.[20] However, this coating process is not suitable for practical applications since dopamine is too expensive. Herein, we

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reported a cheap and simple coating process by using a low cost coating precursor, pyrogallic acid (PA) to replace dopamine, and generated modified PP separators with hydrophilic surface. PA (1,2,3-trihydroxybenzene) has been found in many natural materials such as Eurasian watermilfoil, tannic acid and gallic acid.[21-23] Commercial PA can be obtained from plants. PA has been widely used in many industries such as pharmaceuticals, plastics and cosmetics, and functions as an antiseptic compound, antioxidant and dying material.[23-25] The cost of PA is much lower than that of dopamine (see Table 1). In our experiments, PA was used as the sole coating precursor, and the PA coatings can be formed spontaneously onto the PP separator surfaces in a bis-Tris buffer aqueous solution at room temperature. An enhanced electrochemical performance of LIBs including discharge specific capacity, cycling stability and rate capability was obtained by using the PA coated PP separators. This coating process not only retains many of the advantages of polydopamine coatings but also is a "green and cheap" modification technology due to pyrogallic acid being much less costly than dopamine. To the best of our knowledge, however, this new coating process, based on PA to modify the commercial polyolefin separators for LIBs, has never been reported.

Experimental

Materials

Pyrogallic acid and 2,2-Bis(hydroxymethyl)-2,2',2''nitilotrienthanol (bis-Tris) were purchased from Aladdin Industrial Corporation. PP separators (Celgard 2400) were purchased from Celgard Company. Battery-grade ethylene carbonate (EC), dimethyl carbonate (DMC) and $LiPF_6$ were purchased from Shenzhen Capchem Chemicals Co., Ltd., China, and used without further purificacleantion. $LiMn_2O_4$ cathode materials were purchased from Hunan Reshine New Material Co., Ltd., China. The liquid electrolyte of 1 M $LiPF_6$ in a 1:1 (*wt*: *wt*) EC/DMC used in our experiments was prepared in an argon-filled glove box, in which both oxygen and water content were less than 1 ppm.

Preparation of Pyrogallic acid coating PP separators

PP separators were first rinsed with isopropyl alcohol and then dried at 60 °C for 12 h. After that, the clean PP separators were immersed in a bis-Tris buffer (100 mM buffer and 600 mM NaCl, pH = 7.0) aqueous solution containing pyrogallic acid (1 mg mL^{-1}) at room temperature, and then taken out, washed with deionized water to remove the residual pyrogallic acid. Finally, the resultant separators were dried at 60 °C for 24 h. In our experiments, the separators were immersed in the PA solution for 6 h, 12 h, 18 h and 24 h, which were donated as PA-PP-6, PA-PP-12, PA-PP-18 and PA-PP-24, respectively.

Characterization

Chemical compositions for the separators were analyzed by using Attenuated total reflection-infrared spectra (ATR-IR) with a FT-IR spectrophotometer (Bruker, EQUINOX 55) using a ZnSe crystal at the resolution of 4 cm^{-1} and 64 scans, and X-ray photoelectron spectroscopy (XPS) using a RBD upgraded AXIS ULTRA DLD system with Mg Kα radiation (hγ = 1253.6 eV), respectively. Morphologies of the separators were measured by using field emission scanning electron microscopy (FESEM, JEOL Ltd., Japan). Porosity of the separators was determined by using a gravimetric method with *n*-butanol as pore-filling solvent.[26] The micro-porous structure of the separators was characterized with a Gurley densometer (4110N, Gurley) by measuring the time for air to pass through a determined volume under a given pressure, which was presented by Gurley value (section per 100 cm^3).[27] Water contact angle of the separators was carried out by using a contact angle measuring system (SL200C, USA KINO Industry) at room temperature.

For liquid electrolyte uptake measurements, the separators were soaked in the liquid electrolyte (1M $LiPF_6$ in EC: DMC = 1: 1, *wt: wt*) for 1 h and then taken out. Subsequently, the excessive electrolyte on the separator surfaces was removed away with waterleaf paper prior to measuring the weights. The final uptake values were calculated from the equation: (M2-M1)/M1×100%, where M1 and M2 represent the weights of the samples before and after the immersion in the liquid electrolyte, respectively.

Ionic conductivity of the separators at room temperature was determined with two electrodes AC impedance method by using Autolab PGSTAT302 electrochemical test system (Eco Chemie, the Netherlands) over a frequency range of 10^{-2} to 10^6 Hz.

Electrochemical measurements

The $LiMn_2O_4$ electrodes were prepared by mixing the $LiMn_2O_4$ powder (80 *wt*%), carbon black (10 *wt*%) and PVDF (10 *wt*%) in *N*-methylpyrrolidone (NMP) solvent. The mixed slurry was coated onto aluminum foil, dried at 80 °C for 8 h, and then left at room temperature overnight. The electrode disks were then punched out of the coated foil sheets and weighted. $LiMn_2O_4$/Li half cells were fabricated with 2016-coin type cells in the argon-filled glove box using the PP separators with and without PA coating. The charge-discharge performance of the cells was tested on Land CT2001A tester (Wuhan, China) at the constant current mode over the range of 3.0-4.5 V.

Results and discussion

From Figure 1a, it can be found that the commercial PP separators which are normally manufactured by "dry process"[7] show micro-porous structures with pore diameters of about dozens to hunderds nanometers. Similar to the polydopamine coatings reported in the literature,[20] the PA coatings can also be formed spontaneoulsly on the immersed PP separators in our experiments since the color changed from white to brown (see Figure 1). With increase the immersed time, the color of the PA coated PP separators changed from light brown to dark brown, which indicates that the weight content of the PA coatings on the separators was increased. The maxium weight content of the PA coatings was about 1.8 ± 0.2 *wt*% for the PA-PP-24 separator. The PA coatings were covered on the surface of separators as shown in Figure 1, however, it can be found that the PA coated PP separators still maintain micro-porous structures. In addition, the thickness of the PA coated PP separators are almost similar to that of the original PP separator (detailed data see Table 1). As shown in Figure 2, it can be found that there are only a slightly changes in the thickness of the PA-PP-24 separator (22±0.8 μm) compared with the original PP separator (22±0.2 μm).

Compared with the original PP separator, both porosities and Gurley values of all the PA coated PP separators almost did not change (see Table 1). These results indicate that the PA coatings should be thin enough that the pores inside the separators are not largely hindered.

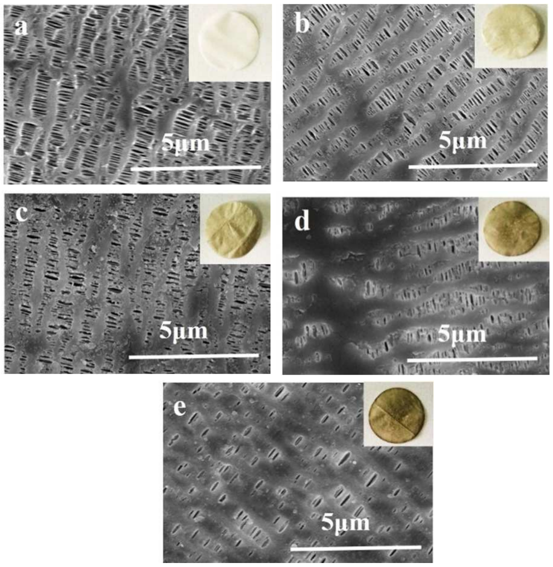


Fig. 1 SEM images of PP separators without (a) and with pyrogallic acid treatment for 6 h (b), 12 h (c), 18 h (d) and 24 h (e).

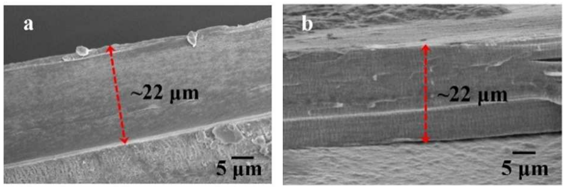


Fig. 2 SEM images of cross-section of PP separators without (a) and with pyrogallic acid treatment for 24 h (b).

Table 1 Physical properties of separators.

Separators	PP	PA-PP-6	PA-PP-12	PA-PP-18	PA-PP-24
PA content / wt%	0	0.4±0.1	0.8±0.1	1.2±0.1	1.8±0.1
Thickness / μm	22±0.2	22±0.2	22±0.3	22±0.5	22±0.8
Porosity / %	42.2	42.2	42.0	41.9	41.0
Gurley value / sec 100cm^{-3}	610	610	618	621	635
Electrolyte uptake / wt%	~92	~101	~114	~123	~129
Ionic conductivity / mS cm^{-1}	0.23	0.31	0.39	0.46	0.57

The PA coatings on the PP separators were further confirmed by ATR-IR and XPS measurements. ATR-IR spectra of the original PP and PA coated PP separators were shown in Figure 3. For the original PP separator, it can be found the characteristic peaks of PP polymers, such as the peak at 2951 cm^{-1} represented the non-symmetric stretching vibration of -CH$_3$, the peak at 2842 cm^{-1} attributed to the symmetric stretching vibration of C-H, and the peaks at 1455 and 1376 cm^{-1} ascribed to the non-symmetric and symmetric bending vibrations of -CH$_3$, respectively.[7,18,19] For the PA coated PP separators, all characteristic peaks originated from the original PP separator are still preserved, while some new peaks appear. A broad peak between 3600 and 3100 cm^{-1} is attributed to the -OH stretching vibration.[28] The peaks at 1627 and 1582 cm^{-1} are ascribed to the C-O stretching vibration and the C=C stretching vibration of aromatic ring, respectively.[28] And the intensity of these new peaks increased with increase the immersed time for the PA coated PP separators. Because the PA weight contents of the PA-PP-18 and PA-PP-24 separators were higher than other PA coated PP separators (see Table 1), two small new characteristic peaks of pyrogallic acid appeared in the the PA-PP-18 and PA-PP-24 separators: the peak at 1117 cm^{-1} ascribed to the asymmetric stretching vibration of C-O-C and C-OH, and the peak at 1040 cm^{-1} attributed to the symmetric vibration of ether bond, respectively.[28] As the XPS spectra shown in Figure 4, the original PP separator mainly shows a C 1s peak at 284.8 eV. After the PA coatings, a new O 1s peak at 532.8 eV appeared in the XPS sepactrum of the PA coated PP separator corresponding to the oxygen-containing groups from the PA molecules. These results indicated that the pyrogallic acid has been successfully coated on the surface of the PP separator.

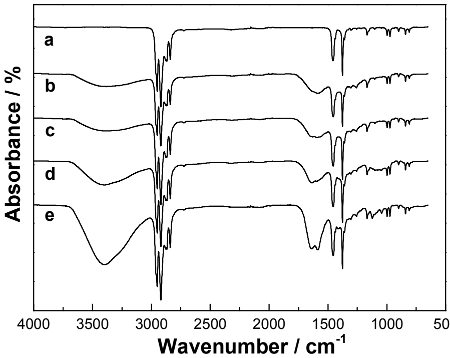


Fig. 3 ATR-FTIR spectra of PP separators without (a) and with pyrogallic acid treatment for 6 h (b), 12 h (c), 18 h (d) and 24 h (e).

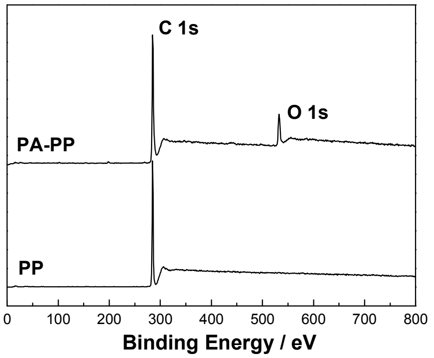
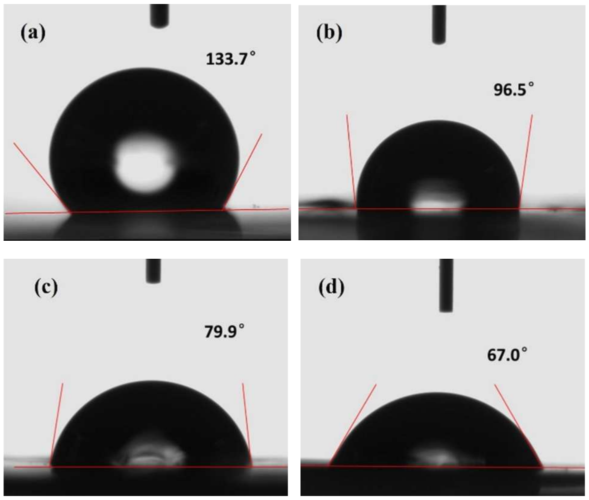


Fig. 4 XPS spectra of PP separators without (PP) and with pyrogallic acid (TA-PP) treatment for 24 h (TA-PP).

The wetting capability of the separators is evaluated by the contact angle measurements with water droplets. As shown in

Figure 5, the original PP separator exhibits hydrophobic wetting behavior, and its water contact angle is about 133.7° (Figure 5a). After the hydrophilic PA coatings, the water contact angles of all the PA coated PP separators were smaller than that of the original PP separator. And the water contact angle decreased with increase the immersed time for the PA coated PP separators. Among all the separators with and without PA coatings, the minimum water contact angel was obtained at about 54.7° for the PA-PP-24 separator in our experiments. These results indicate that the PA coated PP separators become more hydrophilic and show better wetting capability than that of the original PP separator. The wetting capability of the liquid electrolyte solvents on the separators was further carried to test the liquid electrolyte (1M $LiPF_6$ in EC: DMC = 1:1, *wt*: *wt*) uptake of the separators. In general, the better wetting capability of the separator is, the higher liquid electrolyte uptake is.[7,18,19] As expected, the liquid electrolyte uptake increased with increase the immersed time for the PA coated PP separators (detailed data see Table 1). The maximum liquid electrolyte uptake was obtained at about 129 *wt*% for the PA-PP-24 separator due to its best wetting capability among all the separators in our experiments. Generally, ionic conductivity depends on the content and mobility of Li^+ ions,[29] which are mainly determined by the micro-porous structure (porosity and Gurley value) and wetting capability (liquid electrolyte uptake) of the separators in LIBs. From Table 1, it can be found that both porosities and Gurley values of all the PA coated PP separators are not significantly different compared to the original PP separator. This means that the diffusion resistances of Li^+ ions (representing Li^+ mobility) in all the PP separators with and without PA coatings are not significantly different. So the ionic conductivity will mainly depend on the liquid electrolyte uptake (representing Li^+ content). Due to the higher liquid electrolyte uptake of the PA coated PP separators, the ionic conductivities of all the PA coated PP separators were also higher than that of the original PP separator (see Table 1). Among all the separators, the maximum value of the ionic conductivity was about 0.57 mS cm^{-1} for the PA-PP-24 separator, which was about 1.5 times higher than that of the original PP separator (0.23 mS cm^{-1}).



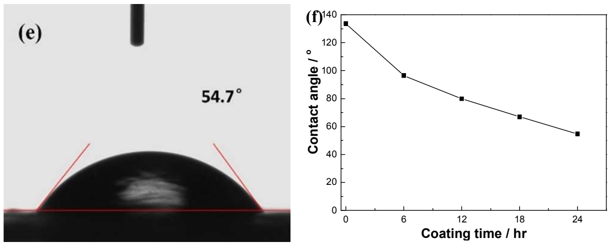


Fig. 5 Water contact angle images of PP separators without (a) and with pyrogallic acid treatment for 6 h (b), 12 h (c), 18 h (d) and 24 h (e). (f) Water contact angle of PP separators as a function of pyrogallic acid coating time.

It is worth noting that our surface coating method is much cheaper than the polydopamine coating method reported in the literature.[20] According to the price of Aladdin, as shown in Table 2, the cost of 1 L of 10 mM dopamine solution (used in the literature)[20] is about \$6.73, however, the cost of 1 L of 1 mg mL^{-1} pyrogallic acid solution (used in our experiments) is only about \$0.14.

Table 2 Cost of reagents.

Material	Dosage in 1L solution	Unit Price [a] (100g)	Total
Dopamine	1.9g	\$354.26	\$6.73
pyrogallic acid	1g	\$14.34	\$0.14

[a] Both the reagents are calculated according to the price in Aladdin.

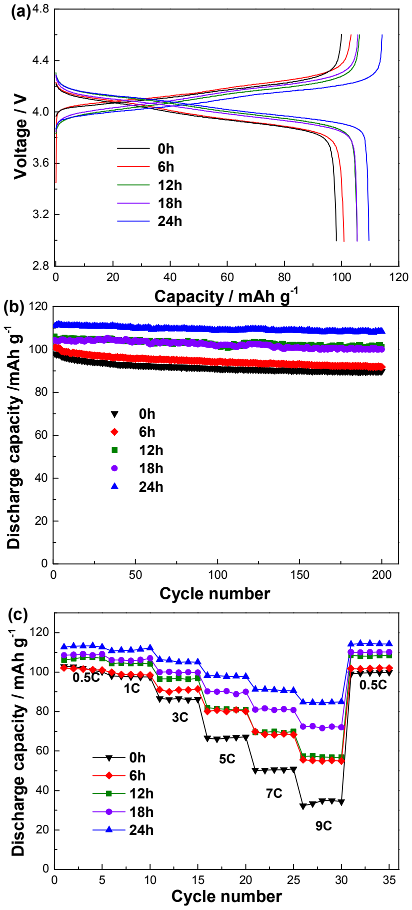


Fig. 6 Electrochemical characterizations for the Li/separator/$LiMn_2O_4$ cells using PP separators with and without pyrogallic acid coating. (a) Charge-discharge profiles at the 1st cycle for the cells with different separators (1 C for both charge and discharge). (b) Cycling stability of different separators. Rate: 1 C. (c) Discharge capacity profiles of the cells at various rates. Charge rate: 0.5 C.

Table 3 Initial electrochemical performance of lithium-ion batteries.

Separators	Initial discharge capacity / mAh g^{-1}	Initial charge capacity / mAh g^{-1}	Coulombic efficiency / %
PP	98.1	100.0	98.1
PA-PP-6	100.8	103.3	97.6
PA-PP-12	105.4	105.7	99.8
PA-PP-18	105.5	106.2	99.2
PA-PP-24	111.8	114.2	97.9

To evaluate the effect of the PA coated PP separators on their electrochemcial properties, spinel $LiMn_2O_4$ (cathode)/Li (anode) half cells with the conventional liquid electrolyte (1M $LiPF_6$ in EC: DMC = 1:1, *wt*: *wt*) were prepared. Figure 6a shows the initial charge-discharge profiles of the cells cycled under a voltage range of 3.0-4.5 V at a current density of 1 C. In these profiles, there are two pseudoplateaus at around 3.9 and 4.1 V, which are ascribed to the typical electrochemical behavior of the spinel $LiMn_2O_4$.[30] Due to the higher liquid electrolyte uptake and higher ionic conductivity of all the PA coated PP separators, both initial discharge and charge capacities of the cells using the PA coated PP separators show a slightly higher than that of the cell using the original PP separator (see Table 3).

The cycle performance of the cells using the original PP and PA coated PP separators are shown in Figure 6b. After 200th cycles at a current density of 1 C, the discharge capacity retention values of all the cells were about 91.6% for the original PP separator, 91.0% for the PA-PP-6 separator, 95.5% for the PA-PP-12 separator, 96.0% for the PA-PP-18 separator and 97.7% for the PA-PP-24 separator, respectively. Most of the PA coated PP separators have higher capacity retention values than that of the original PP separator. And the best cycling stability and the minimum fade in the discharge capacity are found for the PA-PP-24 separator. These results indicate that the PA coated PP separators show better cycling stability than that of the original PP separator.

The good wetting capability of the PA coated PP separators on the rate capability (power performance) was confirmed by testing the discharge specific capacities at the current densities in the range from 0.5 C to 9 C. As clearly shown in Figure 6c, the discharge capacities of the cell using the original PP separator dropped drastically from 102.7 mAh g^{-1} at 0.5 C to 33.3 mAh g^{-1} at 9 C (32.4% of the discharge capacity at 0.5 C). In contrast, the cells using the PA coated PP separators showed higher discharge capacity than that of the cell using the original PP separator at different current densities. And the best power performance is obtained for the PA-PP-24 separator. When the current densities increased from 0.5 C to 9 C, the discharge capacities of the cell using the PA-PP-24 separator changed from 112.6 to 84.7 mAh g^{-1} (75.2% of the discharge capacity at 0.5 C). From these results, we can know that both cycling stability and power performance of the LIBs using the PA coated PP separators were more improved. Maybe the main reason is that the enhanced wetting ability of the PP separator will increase the liquid electrolyte retention during cycling. In addition, the stability of the PA coatings on the PP separators was also evaluated by the XPS characterization. As shown in Figure 7, both original O 1s and C 1s peaks were well preserved for the PA coated PP separator after 200th cycles. This indicates that the PA coatings have good stability and strong adhesion onto the PP separators.

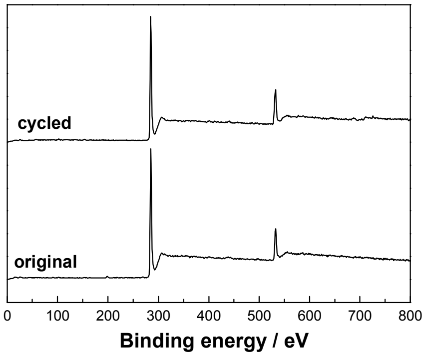


Fig. 7 Stability of pyrogallic acid coating on the PP separators before (original) and after 200th cycles test at 1 C rate in the Li/separator/$LiMn_2O_4$ cells (cycled).

Conclusions

In summary, a cheap and simple dipping surface coating method by using pyrogallic acid as coating precursor was reported to solve the wetting capability issue of commercial polyolefin separators for LIBs. With this method, commercial PP separator can become more hydrophilic and show better wetting capability, which not only improves some critical properties of separators such as electrolyte uptake and ionic conductivity, but also significantly enhances the LIBs performance including discharge specific capacity, cycling performance and power performance. We expect that this "green and cheap" surface coating process is quite versatile and thus is application to develop other separators for energy storage devices (such as lithium-ion battery, supercapacitor and flow battery) or new porous membranes for membranes separation processess.

Acknowledgements

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Pyrogallic acid coated polypropylene membrane as separator for lithium-ion batteries

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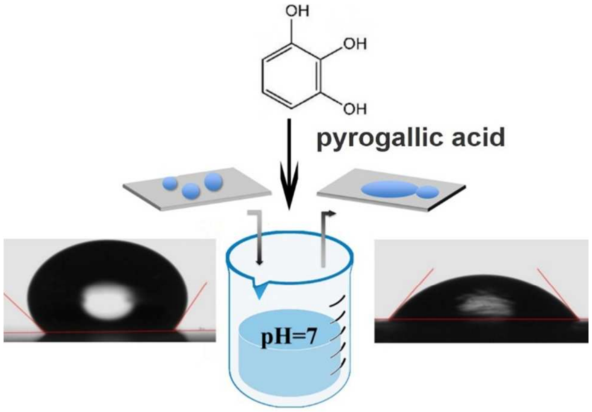
Pyrogallic acid (PA) coated polypropylene (PP) separators for lithium-ion batteries (LIBs) are developed. Due to the PA coating, it makes the PP surfaces hydrophilic and thus improves LIBs performance including specific capacity, cycling stability and power capability compared to the original PP separators.



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